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BALLISTIC COMPRESSOR EXAMINATION OF ELECTRODEPOSITED
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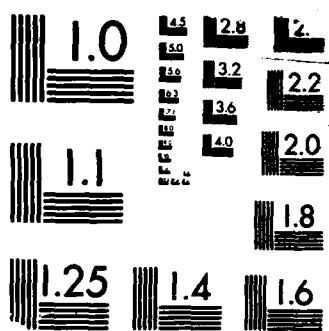
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BALLISTIC COMPRESSOR EXAMINATION OF ELECTRODEPOSITED NIOBIUM

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LAURENCE D. JENNINGS
MATERIALS SCIENCE BRANCH

ALFRED S. MAROTTA
MATERIALS TESTING AND EVALUATION BRANCH

S. KING PAN
BENET WEAPONS LABORATORY

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ABSTRACT

As part of a program to simultaneously examine the capabilities of the MTL ballistic compressor and to characterize materials, a series of high pressure, high temperature shots were made on specimens of niobium electrodeposited on a copper substrate. Each shot allowed the test gas to flow through an experimental channel. The resulting surfaces were examined optically and by scanning electron microscopy, and mass loss was measured. On the basis of these, it was noted that the niobium offered a measure of protection to the underlying material, and underwent a different damage mechanism than does steel. This mechanism is elucidated.

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INTRODUCTION

A "ballistic compressor" is actually a device which uses a compressed driver gas to quasi-adiabatically compress a test gas. As part of a program to both investigate the usefulness of such an instrument, and to establish properties of possible refractory protective surfaces, a series of experiments were carried out on surfaces of niobium electrodeposited on copper. These are described here and a picture of the surface damage process is developed in this report.

THE BALLISTIC COMPRESSOR SHOTS

The MTL ballistic compressor attains gas pressures of the order of 2000 atmospheres and temperatures of the order of 5000 Kelvins. In order to obtain high heat transfer and shearing wipe off conditions simulating those which might occur in a gun tube or rocket nozzle, the hot gases were constrained to flow through a channel about 130 micrometers thick by 3.2 mm wide for a length of about 1.5 mm. One side of this channel consisted of the niobium-plated "test" specimen. Unfortunately it was not feasible to construct the other side of the channel of the same material; instead 4340 steel was used, as sketched in Figure 1.

The test specimens were prepared by electrodeposition from molten fluoride salts in a FLINAK bath.^{1,2} Depositions were carried out at a bath temperature of 800°C with a pulse current of 500 microseconds on, and 500 microseconds off, at a peak density of 40 mA/cm². Specimen A9 was deposited from a solution of NbF₅ dissolved in the FLINAK, whereas A47 was from a 10% NbF₅/10% TaF₅ mixture. These different preparation techniques were used in anticipation of possible different

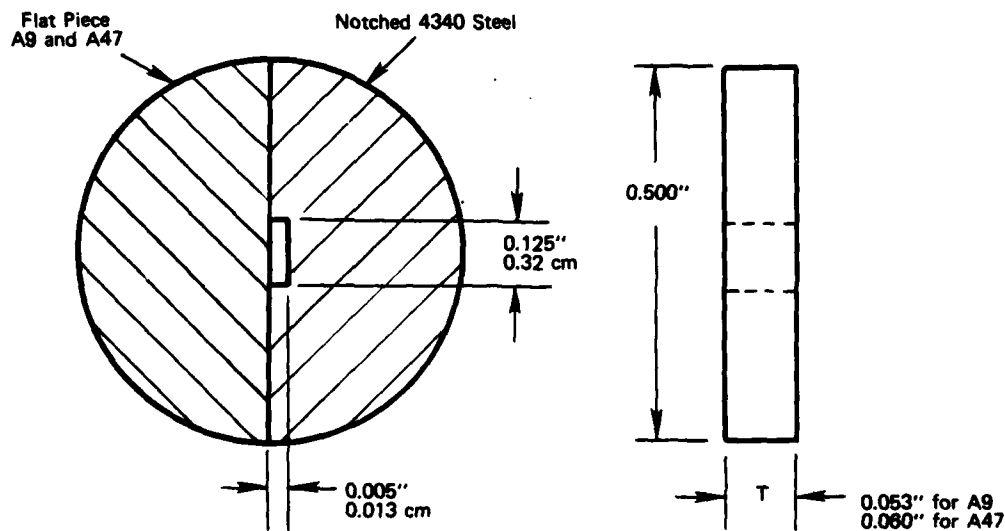


Figure 1. Sample dimensions, showing the narrow erosion/corrosion channel milled in one of the two pieces which make up the disk.

1. LEVY, M., and PAN, S. K. *Refractory Metal Coatings for Control of Gun Tube Erosion*. Proceedings of the Tri-Service Gun Tube Wear and Erosion Symposium, Dover, NJ, 25-27 October 1982, Session IV, p. 354-367.
2. MELLORS, G. W., and SENDEROFF, S. *Electrodeposition of Coherent Deposits of Refractory Metals*. J. Electrochem. Soc., v. 112, no. 3, p. 266-272, 1965.

resultant coatings. However, analysis showed that the deposit was practically pure Nb in either case. Both sides and the edges of each copper substrate were coated. The specimens were prepared by polishing off the coating from the faces of the substrate; these faces then became the faces of the sample disks. The coating on the edge of the substrate was polished to present a flat surface to be tested. The residual coating was of the order of 100 micrometers thick for each specimen.

The test gas was a mixture of 10 percent (by volume) nitrogen in argon, closely approximating 10 percent by molecules. This mixture was chosen because it achieves a temperature adequate for melt and wipe off at pressures appropriate both to gun barrel conditions and to the test section of the MTL ballistic compressor. The half-width of a pressure pulse was typically 1.5 milliseconds for all shots.

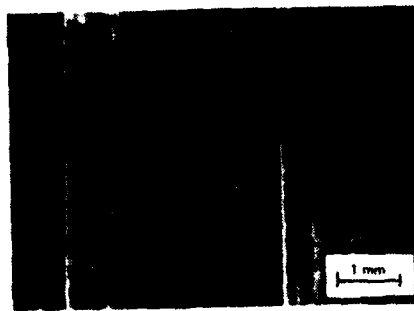
All runs were made with the same initial test gas pressure of 0.5 atmosphere. The runs were made with varying reservoir pressures (P_r), which serve as a source of driving force on the piston. These, in turn, determine the maximum pressure of the compressed test gas (P_{max}). Using the results of Lalos and Hammond,³ the maximum temperature (T_{max}) is estimated as being 25 percent lower than that due to ideal adiabatic pressure with no gas leakage or heat loss to the surroundings.

Before and after each shot, each half of the disc specimen was weighed to an accuracy of about 10 micrograms. (The sensitivity of the balance was greater than this and measurements are reported to one microgram.) Each surface was also photographed before and after each shot. Typical photographs are shown in Figures 2 and 3.

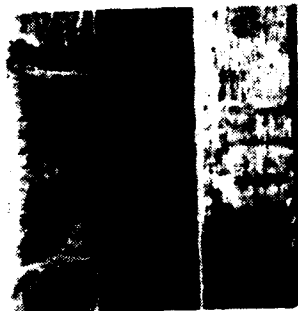
Table 1 is a summary of the seven compressor shots. A series of six shots were made on the specimen consisting of the A9 piece. Shot no. 57, the initial shot in this series, utilized conditions which, experience has shown, lead to appreciable melt and wipe off of a 4340 sample. Indeed, as shown by the results in Table 1, there was significant mass loss from the 4340 portion of the sample. The A9 portion of the sample was inappreciably affected, as seen in Figure 2. To put the mass loss for the 4340 in perspective, it may be noted that 140 micrograms represents a thickness of about 4 micrometers, about 3 percent of the initial channel thickness of 130 micrometers. Thus, subsequent shots (under increasingly severe conditions) were through a channel which was only changed slightly from the original, although the total change through the series was about 17 percent of the original thickness. Our experience has been that the 4340 mass loss under increasingly severe conditions is not as great as might have been expected. This conclusion is verified by the fact that the average mass loss in shots 58 to 62 was no more than in the initial shot. The same conclusion was verified in another way by shot no. 63 on an unprotected, virgin piece of 4340 steel. In this case, the loss to the 4340 in one shot was comparable to the total loss in the six shots on the 4340 paired with A9.

The increasingly severe conditions did have some effect, noticeable at low magnification, on sample A9, as shown in Figures 2 and 3. These effects were accompanied by no significant mass loss. There are small indications of discoloration due to chemical effects and a rippled pattern arising from the flow

3. LALOS, G. T., and HAMMOND, G. L. *The Ballistic Compressor and High Temperature Properties of Dense Gases* in *Experimental Thermodynamics, Volume II, Experimental Thermodynamics of Non-Reacting Fluids*, B. LeNeindre and B. Vodar, ed., Butterworths, London, 1975, p. 1193-1218.



Virgin Samples Prior to Shot No. 57



Shot No. 57



Shot No. 58



Shot No. 59



Shot No. 60



Shot No. 61



Shot No. 62

Figure 2. Low magnification photographs of paired samples 4340 steel and niobium-coated A9. In each photo the 4340 is on the left, while A9 is on the right. Direction of gas flow is from left to right. Shot conditions are listed in Table 1.



Virgin Samples Prior to Shot No. 63



Shot No. 63

Figure 3. Sample A47 with its paired 4340 steel before and after the shot.
Other particulars are the same as Figure 2.

Table 1. RESULTS OF COMPRESSOR SHOTS

Date	Shot No.	Pr (atm)	Pmax (atm)	Tmax (Kelvin)	Mass Change (ug)	
					Notch(GS)*	Flat(A9)
12/6/84	57	22.40	1703	4986	-140	-20
12/13/84	58	20.70	1736	5022	-136	-19
12/18/84	59	21.03	1753	5041	- 49	+14
12/21/84	60	21.52	1840	5136	-100	+ 1
12/28/84	61	22.00	1992	5294	-233	+ 4
1/4/85	62	22.62	2162	5463	- 65	- 8
TOTAL MASS CHANGE					-723	-28
					Notch(GS)*	Flat(A47)
1/10/85	63	22.62	2078	5381	-560	+ 8

*GS (gun steel) refers to the 4340 half-discs.

behavior of the moving gases. The single shot on A47 showed more discoloration than was visible on A9 after the series of six shots. Furthermore, there was more evidence of disruption of the surface, perhaps arising from deposits connected with the large mass loss in the 4340 steel. As previously stated, the mass change in A47 was also negligible.

A significant result of the experiment is that the niobium-plated material, though not unaffected by the hot gas flow, suffered neither appreciable melt and wipe off as did the steel nor any flake off, which might have arisen from poor bonding to the substrate. Repeated exposures to the erosion conditions did, however, give rise to observable cracking, as discussed below.

Additional analysis, using Auger electrons for example, would clearly be desirable, but could not be carried out with the resources available to the present project.

SCANNING ELECTRON MICROSCOPY (SEM) EXAMINATION

After all shots were made, SEM micrographs were taken of the two niobium-plated samples. The A9 had been subjected to a series of shots of increasing intensity, whereas A47 was subjected to a single shot comparable in intensity to the last shot on A9. Although the preparation conditions for the two samples were different, we assume, based on the measured compositions, that the coatings were substantially the same.

In most of the micrographs the marks left by the polishing are plainly visible. These serve as valuable references and are helpful in explaining the processes which took place during the firings. In all cases the gas flow was from the bottom to the top of the micrographs. The location of each micrograph on the sample is indicated in Figure 4, the micrographs themselves being given in Figures 5 and 6 (reversed from left to right from the actual situation shown in Figures 2 through 4).

The single shot on A47 yielded more visible discoloration than did the totality of shots on A9. This effect is also seen in micrograph 1 which shows the interface between the damaged and undamaged regions of the sample. (The damaged region, which appears light and is on the right of the micrograph, is that which appears discolored to the naked eye.) The explanation is offered by micrographs 2 and 4, which show this interface at higher magnification. Note that the damage (on the right side) is not sufficient to obliterate the polishing marks, but that the surface is noticeably disturbed. This disturbance is also shown in micrograph 5 where the leading edge is noticeably more swirled than is the trailing edge. Thus, we note that the swirling is more conspicuous at the leading edge and along the boundaries of the channel than it is in the central and trailing portions of the channel. Although some damage was evident, to a greater or lesser degree, everywhere on the exposed surface, no cracking of the niobium was observed.

The series of shots on A9 had quite a different effect resulting in an appreciably different appearance to the naked eye, as well as to the SEM. The basic difference is that the most conspicuous swirling was in the central and trailing portions of the channel. Micrograph 11, taken at the same magnification as micrograph 1, shows the conspicuous feature at the upper left marking the boundary of the channel. Note that the swirling is much reduced in just the regions where it was

greatest in A47. On the other hand, in the central portion (represented by the upper right hand portion of micrograph 11) where the swirling is greatest in A9, this swirling is more evident, even at Mag. 100X, than is the swirling in micrograph 1. This general behavior, that the entrance surface of A47 resembles the exit region of A9, is confirmed by micrograph 10. Here we see an appearance more like micrograph 4 (of the leading edge) than like micrograph 2 (of the trailing edge). Note that the region of reduced swirling (on the left in micrograph 10) is still well within the boundaries of the channel.

The origin of the swirling is further defined by micrograph 13 taken away from the damaged region, 14 taken at the leading edge, and 15 taken at the trailing edge, all at high magnification. The polishing marks are readily visible in 13, are somewhat obscured in 14 and are essentially invisible in 15. However, 15 suggests that the polishing marks have been covered over, especially in view of the obvious covering over of the cracks by a deposit.

Quite a number of these cracks were observed in A9. Micrograph 15 suggests that these cracks appear after some of the deposit was laid down, but before the remainder of the deposit was laid down. Thus, we conclude that the cracks occurred during one or more of the firings of the ballistic compressor. This is confirmed by micrograph 16, which shows a crack under high magnification. Several features formed during an early shot clearly bridge the gap of the crack.

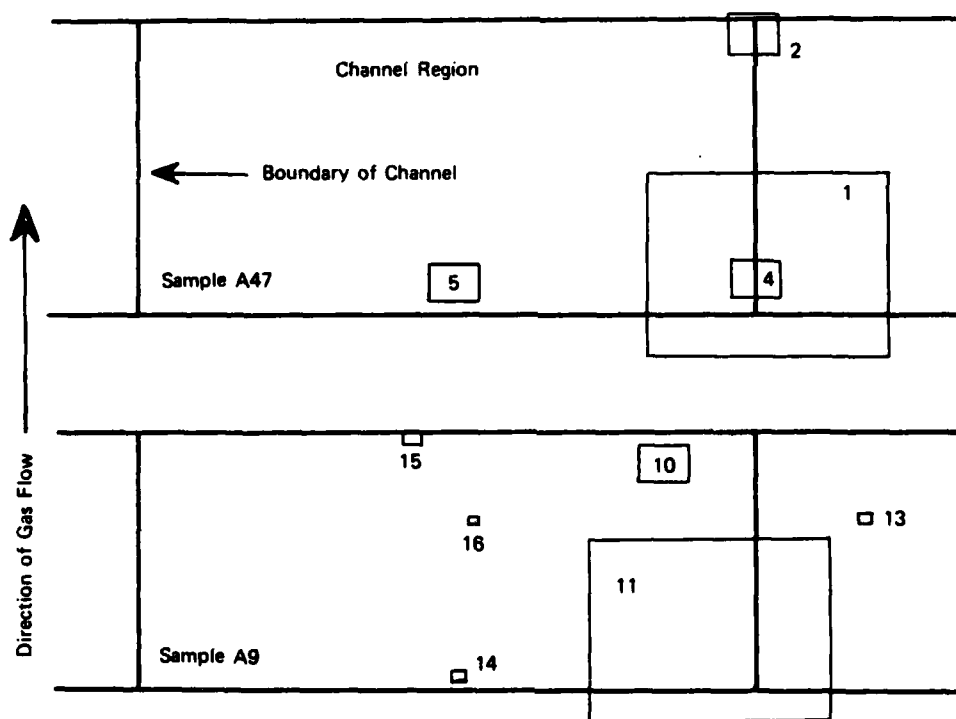
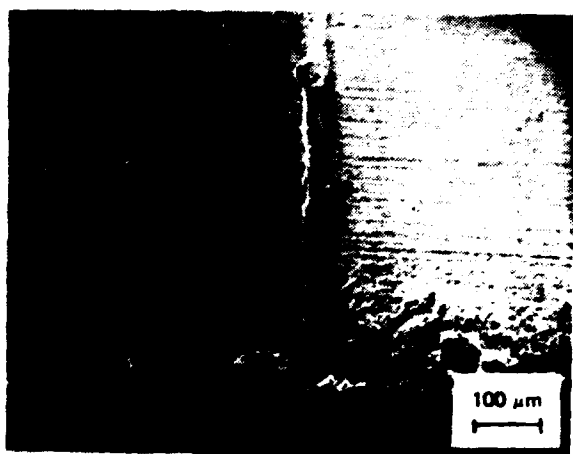
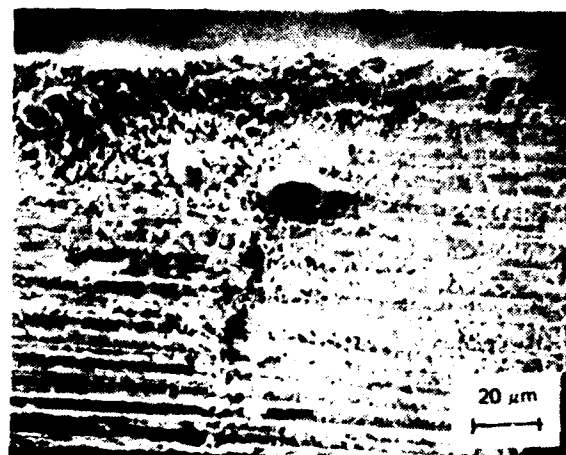


Figure 4. Location of selected SEM micrographs in relation to channel regions of A9 and A47. The numbering scheme identifies the actual micrographs along with their magnifications as shown in the succeeding Figures 5 and 6.



Micrograph 1 Mag. 75X



Micrograph 2 Mag. 375X

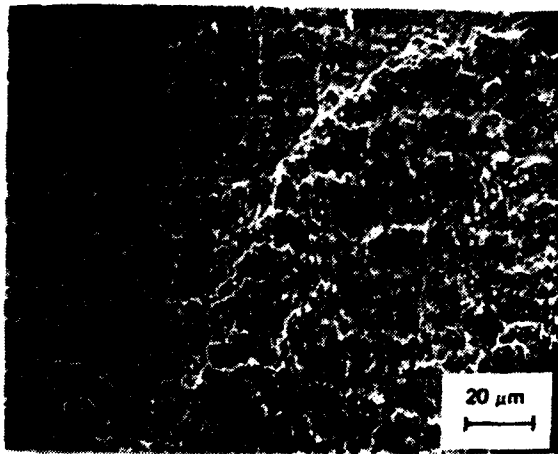


Micrograph 4 Mag. 375X

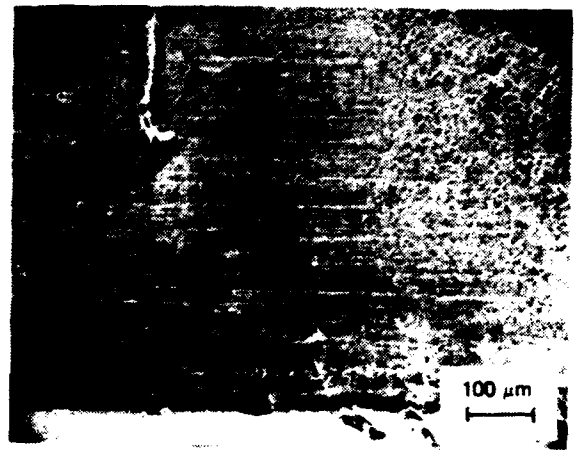


Micrograph 5 Mag. 375X

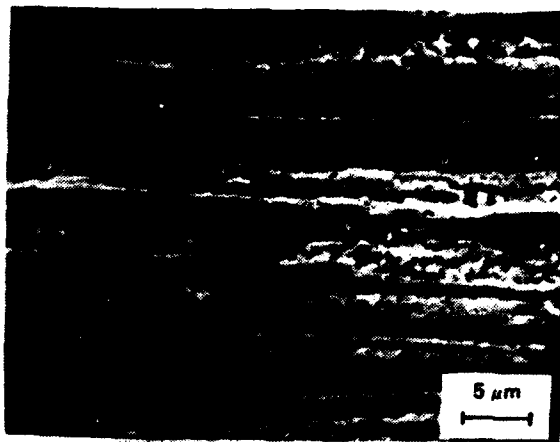
Figure 5. SEM micrographs of niobium-coated sample A47. Direction of gas flow is from bottom to top.



Micrograph 10 Mag. 375X



Micrograph 11 Mag. 75X



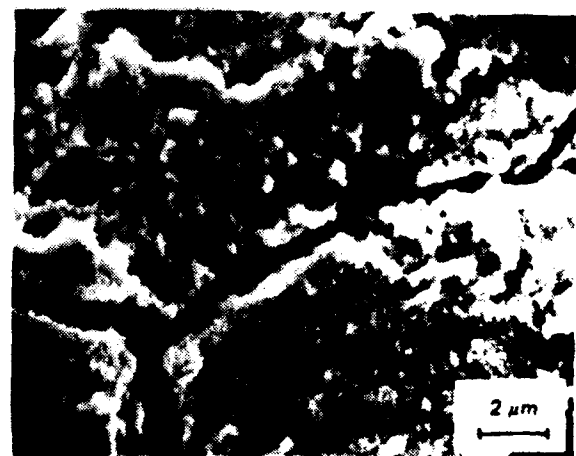
Micrograph 13 Mag. 1500X



Micrograph 14 Mag. 1500X



Micrograph 15 Mag. 1500X



Micrograph 16 Mag. 3750X

Figure 6. SEM micrographs of niobium-coated sample A9. Direction of gas flow is from bottom to top.

CONCLUSIONS

From all these results, we obtain the following picture of the damage process, which takes place with negligible mass removal in the present instance. The first stage of damage is a swirling of the surface, leaving the polishing marks more or less intact. Further damage removes this swirling from some portions of the sample, but results in the deposited appearance, shown in micrograph 15, elsewhere in the sample. This effect, which we have termed a redeposit, completely obliterates the polishing marks. The most affected parts of a channel are the leading edge and the side boundaries. In a single shot there is the greatest disturbance of the surface in these areas. In a multiple shot situation, the disturbance is wiped off in these regions and redeposited in the central and trailing sections. This leaves the leading and boundary portions looking more like their original appearance than does a single shot. The redeposited region, on the other hand, is far different than the original. Furthermore, the repeated firings lead to cracking of niobium plated on copper.

Although the Nb coating underwent the damage just described, it is noteworthy that the mass loss to the Nb was far less than to 4340 steel under similar conditions.

These conclusions show that the ballistic compressor can be used to produce interesting effects on refractory materials. Much more work would be required to make statements about the comparative suitability of such refractory surfaces in applications.

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2	ATTN: SLCMT-IML
3	Authors

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BALLISTIC COMPRESSOR EXAMINATION OF
ELECTRODEPOSITED NIOBIUM
Laurence D. Jennings, Alfred S. Marotta,
and S. King Pan

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